

## An Electrical Method for Measuring Fatigue and Tensile Properties of Thin Films on Substrates

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**Keywords:** Basquin equation, electrical testing, fatigue, interconnect reliability, thermal cycling, thermal fatigue, thin-film fatigue, thin-film strength

**Abstract.** A novel approach for measuring thermal fatigue lifetime and ultimate strength of patterned thin films on substrates is presented. The method is based on controlled application of cyclic joule heating by means of low-frequency, high-density alternating current. Such conditions preclude electromigration, but cause cyclic strains due to mismatch in coefficients of thermal expansion between film and substrate. Strain and stress are determined from measurement of temperature. Fatigue properties are a natural fit to testing by alternating current. Stress-lifetime (S-N) data were obtained from patterned aluminum lines, where stress amplitude was varied by changing current density, and lifetimes were defined by open circuit failure. Electron microscopy and electron backscatter diffraction observations of damage induced by a.c. testing suggested that deformation took place by dislocation mechanisms. We also observed rapid growth of grains – the mean diameter increased by more than 70 % after a cycling time of less than six minutes – which we attribute to strain-induced boundary migration. Ultimate strength was determined by extrapolating a modified Basquin relation for high cycle data to a single load reversal. A strength estimate of  $250 \pm 40$  MPa was determined based on a.c. thermal fatigue data. In principle, an electrical approach allows for testing of patterned films of any dimension, provided electrical access is available. Furthermore, structures buried beneath other layers of materials can be tested.

### Introduction

We describe an electrical test method for measuring thermal fatigue lifetime [1] and ultimate strength [2] of thin films and interconnects on silicon substrates. Such an approach circumvents some of the difficulties associated with microtensile testing of free-standing films [3] or nanoindentation of films on substrates [4]. In terms of implementation and interpretation, nontrivial specimen preparation or nonstraightforward property extraction tend to render such methods cumbersome in a manufacturing environment.

Thermomechanical fatigue is a reliability concern in any multimaterial system that undergoes significant thermal cycling, because strains due to differential thermal expansion between the materials can accumulate during manufacturing or operation. Thermal strains of approximately 0.1 % can result from a 70 °C temperature change, which is a typical temperature excursion for a CPU during processor-intensive usage [1]. Such a strain corresponds to stresses of 100 MPa for Al on Si and 125 MPa for Cu on Si, both of which are comparable to thin film yield strengths as measured by microtensile testing.

The reliability concern due to thermomechanical fatigue centers on failure initiation following accumulation of strain during cycling. Failures can take the form of increases in resistivity due to introduction of excessive numbers of lattice defects [5], brittle film cracking due to deformation of underlying metal films [6], and even open or short circuits due to metal deformation [7]. While devices to date have not exhibited significant numbers of failures due to such causes (presumably due to the presence of rigid dielectrics, which can suppress surface damage), the incorporation of more

compliant dielectrics such as polymeric low-k materials could become problematic, as plasticity within metal lines would no longer be suppressed [7].

In advanced interconnect architectures, strength is an important design parameter, particularly for generally brittle physical-vapor-deposited metal films, which can fail with little warning. While methods such as microtensile testing and nanoindentation are suitable for measuring strength of films in special configurations, they are limited in their applicability to extremely narrow structures, and are not applicable to buried structures.

A test method that can address the fatigue problem as well as strength measurement in narrow, buried lines adds value in the context of design and performance prediction of new interconnect/dielectric systems in their near-end-use configuration. It can also aid in the development of accelerated tests for reliability assessment. Our goal is to develop such a test based on electrical methods and to qualify it through rigorous comparison to the reference techniques of microtensile testing and nanoindentation.

### An Electrical Method for Controlled Joule Heating

Alternating-current-based tests, under conditions of low frequency and high current density, offer an alternative, promising method for conducting evaluations of thermomechanical fatigue in small-scale structures in both the low- and high-cycle regimes [1,8]. We have also shown that this approach can be used to measure the ultimate strength of such structures [2]. The tests are based on the principle that within each half cycle of current, joule heating of the current-carrying segment causes not only that segment, but also the immediately surrounding materials to heat up. Heat flow in materials that typically comprise a microelectronic device is rapid enough to allow for nearly complete dissipation by the start of the next power cycle, at frequencies of several hundred to several thousand Hertz. Calibrations of resistivity against temperature then allow for a reasonable estimate of the cyclic temperature,  $\Delta T$ , which can be used to estimate cyclic strain,  $\Delta \epsilon$ , and therefore cyclic stress through the relation

$$\Delta \epsilon = \Delta \alpha \cdot \Delta T, \quad (1)$$

where  $\Delta \alpha$  is the difference in coefficients of thermal expansion between the metal and the substrate.

This is the only method so far developed that is capable of testing thermomechanical fatigue behavior of small-scale structures to large numbers of cycles in the materials' in-use conditions, *e.g.*, patterned thin film metallizations on a silicon-based substrate, in either a passivated or unpassivated state. Figure 1 shows schematically how the cyclic dissipation of heat leads to thermomechanical fatigue, where  $j$  = current density,  $T$  = temperature, and  $\sigma$  = stress.

In terms of fatigue testing, the mechanical stress amplitude depends on the magnitude of the cyclic current density. The ratio of the minimum to the maximum stresses, *i.e.*, the R-ratio, can be varied by controlling the rms temperature of the test specimen through substrate heating or cooling. This allows for tests encompassing tension-tension, compression-compression, or fully reversed tension-compression loading. Analyses of the temperature dependence of cyclic current flow and the frequency dependence of cyclic heat flow have been given [8].

Cyclic joule heating can also be used to evaluate properties such as ultimate strength. Fatigue lifetimes and monotonic tensile properties can be correlated through the Basquin relation:

$$\sigma_a = \sigma'_f \left( 2N_f \right)^b, \quad (2)$$

where  $\sigma_a$  is the stress amplitude,  $N_f$  is the number of reversals to failure,  $\sigma'_f$  is the fatigue strength

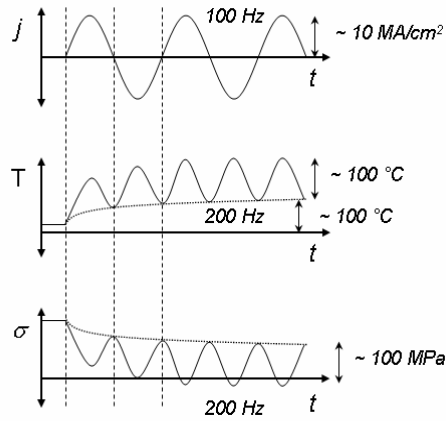


FIGURE 1. Schematic showing relation between low-frequency, high-density alternating current input into an interconnect and resulting temperature and mechanical stress behaviors as a function of time. Values for current density, temperature, and stress are order of magnitude examples.

coefficient, and  $b$  is the fatigue strength exponent.  $\sigma'_f$  is approximately equal to the true fracture strength as determined in a monotonic tensile test [9]. For negligible necking, the Basquin strength coefficient can be approximated by the ultimate tensile strength [10]. This value can be determined by performing several fatigue tests at various stress amplitudes, measuring mean stress and lifetime for each test, and performing a regression analysis. This effectively extrapolates the data back to a single load reversal. This application of the Basquin relation is converse to the originally intended use, which was to estimate cyclic properties based upon knowledge of monotonic tensile properties.

## Experimental

Fatigue tests were carried out on nineteen nonpassivated, single-level structures composed primarily of patterned and etched Al-1 wt. % Si lines sputtered onto thermally oxidized silicon. The lines were 800  $\mu\text{m}$  long, 3.3  $\mu\text{m}$  wide, and 0.5  $\mu\text{m}$  thick. We carried out an additional five tests on nonpassivated, sputtered Cu lines of length 800  $\mu\text{m}$ , width 2.0  $\mu\text{m}$ , and thickness 0.7  $\mu\text{m}$ . Strength tests were carried out on sixteen nonpassivated, single-level structures composed of patterned and etched Al (99.999 % pure) thermally evaporated onto oxidized silicon. These lines were 380  $\mu\text{m}$  long, 5.7  $\mu\text{m}$  wide, and 1.9  $\mu\text{m}$  thick. Current pads and voltage taps were present at each end of every line.

Testing was performed on a four-point probe system using 100 Hz sinusoidal alternating currents with nominally zero d.c. offset. Tests were run in current control, nominally at 20 °C, with simultaneous measurement of resistance. Current densities (rms) in the approximate range (11 to 16)  $\text{MA}/\text{cm}^2$  were applied. Test chips were held in place on a steel stage during testing by use of a vacuum chuck. Lifetime was defined by number of power cycles needed to cause open circuit.

Evolution of microstructure on several Al-1Si lines was documented by use of field-emission scanning electron microscopy (FE-SEM) and automated electron backscatter diffraction (EBSD). Transmission electron microscopy (TEM) was used to observe defect arrangements. In the microstructure example shown in this paper, one line was first characterized by FE-SEM and EBSD prior to testing, in order to record the as-deposited surface and grain structure. The line was then subjected to testing for 10 s and removed from the probe station for another series of FE-SEM and EBSD measurements. Although the test-observe cycle was repeated several times on the same specimen, we present only the microstructure results after an accumulated time of 320 s; refer to [11] for further details.

## Results and Discussion

**Thermal Fatigue Testing.** A plot of rms current density versus lifetime for the Al-1Si lines is shown in figure 2, revealing behavior typical of that represented in fatigue S-N curves. Namely, higher

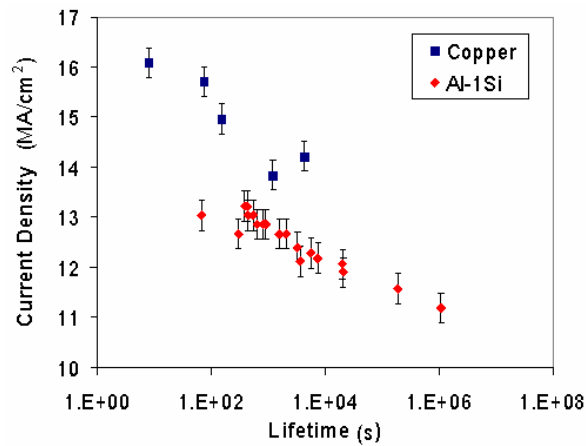


FIGURE 2. Current density versus lifetime for Al-1Si and Cu lines tested by use of alternating current at 100 Hz. Vertical bars represent 0.3 MA/cm<sup>2</sup> uncertainties in current density.

applied stress amplitude (current density, in this case) resulted in lower measured lifetime. Copper lines were able to withstand higher current densities than Al-1Si lines, for a given lifetime. Alternatively, for a given current density, copper showed increased lifetime. All specimens developed damage in the form of surface intrusions and extrusions. Figure 3 shows two sets of FE-SEM/EBSD images from Al-1Si, depicting the changes in both surface topography and grain structure, upon cycling for 320 s, or  $6.4 \times 10^4$  power cycles, at a current density of 12.2 MA/cm<sup>2</sup>. The images were obtained from the same region of the line before and after cycling, and show the heterogeneous nature of damage formation in terms of intrusions/extrusions and grain growth. The selectively occurring damage is attributable to variations in dislocation activity from grain to grain due to effects of crystal orientation and grain size. TEM showed that grains that developed significant intrusions and extrusions contained very few remaining dislocation segments, since most had left the surface to form the topography. Grains that remained relatively flat and intrusion/extrusion-free contained a higher density of remaining dislocation segments.

The mean grain diameter increased by more than 70 % after 320 s of cycling, as determined by EBSD mapping, from 1.4  $\mu$ m to 2.4  $\mu$ m, with an error of less than 10 %. EBSD showed that grains that preferentially grew could be traced back to certain grains in the original microstructure, suggesting that recrystallization did not take place. Grains that showed more surface damage tended also to be the ones that grew. Since those grains contained relatively few remaining dislocation segments, it became clear that this form of grain growth was consistent with strain-induced boundary

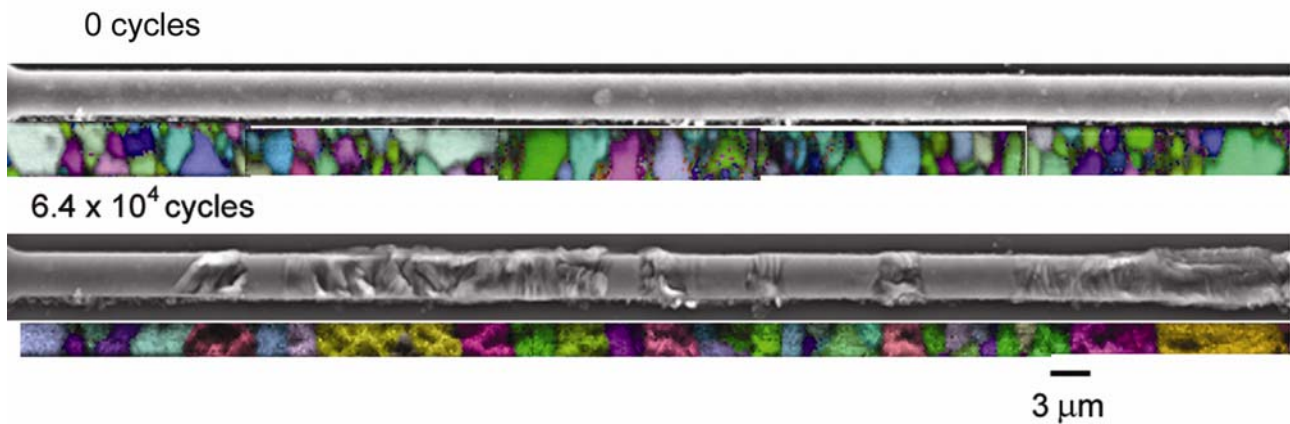


FIGURE 3. Set of FE-SEM/EBSD results showing same region of an Al-1Si line before (top) and after (bottom)  $6.4 \times 10^4$  power cycles at a current density of 12.2 MA/cm<sup>2</sup>. FE-SEM images show formation of intrusions/extrusions. EBSD maps show changes in grain size and orientation.

migration [11,12]. This mechanism describes selective grain growth wherein grains containing significant *residual* plasticity are consumed by those with lower residual plasticity, *i.e.*, strain-energy

minimization as controlled by dislocation content. It tends to overwhelm normal grain growth, which is driven by minimization of surface and interface energies.

**Ultimate Strength Testing.** The tests presented in this paper were applied to sputtered and evaporated metal films, which tend to exhibit residual tensile stresses after deposition. During cyclic joule heating of such structures by alternating current, stress cycles 180° out of phase with temperature, as shown in figure 1. With no control over substrate temperature, stress decreases as applied temperature approaches the deposition temperature near the peak of the power cycle. As the power decreases to zero, stress increases back toward the initial value of the residual tensile deposition stress, neglecting a slight decrease in time-averaged stress due to the slight temperature rise upon reaching steady state. Therefore, during alternating current cycling, in the absence of substrate temperature control, the mean stress will remain tensile. This plays an important role in the application of the Basquin equation, as described next.

A modified form of equation (2) must be used to take into consideration the fact that a nonzero mean stress,  $\sigma_m$ , is present during the course of the test [2,13]. The modified equation is given by

$$\sigma_a = (\sigma'_f - \sigma_m) (2N_f)^b, \quad (3)$$

with other terms as defined earlier. Regression analysis of equation (3) for sixteen specimens, with mean stress taken into consideration, resulted in a fatigue strength coefficient of  $250 \pm 40$  MPa, which compares with an ultimate strength measurement of  $239 \pm 4$  MPa as determined by microtensile testing performed on specimens fabricated from the same wafer [2]. The agreement between these two types of tests of ultimate strength is surprisingly strong, given the dramatically different approaches.

Differences between results obtained by these two test methods may be due to several factors, including effects of substrate constraint and the role of higher temperatures on plasticity and fracture. In the former case, the silicon substrate can impart an increase to the measured strength by providing additional mechanical constraint on dislocation glide [14]. In the case of the effects of high temperature, more plasticity than that typically occurring at room temperature may take place near the peak of the power cycle, as glide and cross slip become easier. As a result, increased strain hardening may then take place during the lower temperature/higher stress portions of the power cycles.

## Summary

Electrical measurements of thermal fatigue lifetime and ultimate strength were made on patterned aluminum and copper interconnects. This method for evaluating mechanical response is based on application of low-frequency, high-density current to effect controlled cyclic joule heating. Interconnect response was consistent with plasticity that develops during fatigue, as determined by SEM, EBSD, and TEM. We observed the formation of intrusions/extrusions and significant grain growth, both of which exhibited considerable site selectivity within the microstructure. The process was consistent with variations in dislocation activity from grain to grain. Grains that showed more intrusions and extrusions grew at the expense of those that remained relatively unchanged, but which contained higher densities of residual dislocation plasticity. This form of grain growth was consistent with strain-induced boundary migration. Extrapolation of fatigue response back to a single load reversal was accomplished through use of a modified Basquin equation, which takes into account the effect of mean tensile stresses present during the course of these tests. This resulted in an estimate of ultimate strength that was consistent to a first approximation with values measured by microtensile testing.

## Acknowledgements

We thank the NIST Office of Microelectronics Programs and the National Research Council Post-Doctoral Associateship Program for support. This work is a contribution of the U. S. Department of Commerce and is not subject to copyright in the U. S. A.

## References

- [1] R. R. Keller, R. Geiss, Y.-W. Cheng, and D. T. Read, in: *Characterization and Metrology for ULSI Technology 2005*, edited by D. G. Seiler, A. C. Diebold, R. McDonald, C. R. Ayre, R. P. Khosla, S. Zollner, and E. M. Secula, American Institute of Physics Conf. Proc. Volume 788, New York (2005), p. 491
- [2] Barbosa III, N., Keller, R. R., Read, D. T., Geiss, R. H., and Vinci, R. P.: *Met. Mater. Trans.* (2006), submitted.
- [3] Brotzen, F. R.: *Int. Mater. Rev.* Vol. 39 (1994), p. 24
- [4] Nix, W. D.: *Mater. Sci. Eng.* Vol. A234 (1997), p. 37
- [5] F. R. N. Nabarro: *Theory of Crystal Dislocations* (Dover Publications, New York 1987), p. 618.
- [6] Huang, A., Suo, Z., and Ma, Q.: *J. Mech. Phys. Sol.* Vol. 50 (2002), p. 1079
- [7] R. R. Keller, R. Monig, C. A. Volkert, E. Arzt, R. Schwaiger, and O. Kraft, in: *Stress-Induced Phenomena in Metallizations: Sixth International Workshop*, edited by S. P. Baker, M. A. Korhonen, E. Arzt, and P. S. Ho, American Institute of Physics Conf. Proc. Volume 612, (2002), p. 119
- [8] Mönig, R., Keller, R. R., and Volkert, C. A.: *Rev. Sci. Instr.* Vol. 75 (2004), p. 4997
- [9] S. Suresh: *Fatigue of Materials* (University Press, Cambridge 1998), p. 223.
- [10] S. S. Manson and G. R. Halford: *Fatigue and Durability of Structural Materials* (ASM International, Materials Park 2006), p. 45.
- [11] Keller, R. R., Geiss, R. H., Barbosa III, N., Slifka, A. J., and Read, D. T.: *Met. Mater. Trans.* (2006), in press.
- [12] Beck, P. A. and Sperry, P. R.: *J. Appl. Phys.* Vol. 21 (1950), p. 150
- [13] J. D. Morrow: *Fatigue Design Handbook - Advances in Engineering* (Society of Automotive Engineers, Warrendale, PA 1968), p. 21.
- [14] Nix, W. D.: *Met. Trans. A* Vol. 20A (1989), p. 2217